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The Effect of Shortening Stability on Commercially Produced Army Ration Biscuits. I. Initial Data and Results of Accelerated Stability Tests*

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DURING World War II the Army purchased millions of pounds of biscuits for use in assembled rations. Since these biscuits were often stored in warehouses and food dumps at elevated temperatures and for periods in excess of one year, it was necessary to use a shortening with maximum stability to insure an edible product, free from rancidity and other undesirable flavors. With the cooperative effort of several industrial groups specifications were hurriedly drawn up. The Swift's Active Oxygen Method (6) for accelerating rancidity in fats and oils was adopted as a specification control procedure, and a minimum stability value of 100 hours was established for the biscuit shortening.

Production of exceedingly stable shortening offered little difficulty to the shortening manufacturer; however, the processor of the biscuit occasionally encountered difficulties in blending the harder shortenings at normal mixing temperatures.

Information was not available as to the significance of the established accelerated stability value of the shortening in terms of actual storage life of the biscuits. Laboratory tests indicated that 100-hour shortening would produce a biscuit of satisfactory storage life, but field observations did not always support the findings. Certain lots of biscuits were found to be rancid approximately one month after manufacture; other lots were quite acceptable after a year or more.

Shortage of vegetable oils of proven quality presented another problem. Approved oils available in important quantities, cottonseed and peanut, became scarce. Before risking the arbitrary lowering of standards, it seemed advisable to further investigate shortenings which had previously been considered unsatisfactory for use in Army biscuits.

In view of the foregoing difficulties a project was set up with the following broad objectives:

1. Determine the relationship between the accelerated stability values (A.O.M.) obtained on the shortening and the storage life of Army ration biscuits.
2. Determine the effect of a phenolic antioxidant on the stability of ration biscuits containing lard.
3. Evaluate the degree and seriousness of "reverted" flavors in ration biscuits containing hydrogenated soybean oil shortening.³

In addition, information was to be obtained relative to the effects of various types of packaging, storage temperatures, and types of ration biscuits. This paper, the first of two, presents the plan of the project, initial analytical data, and evaluations of the

accelerated stability tests. Results of the two-year storage study on development of oxidation will be presented in the second paper.

TABLE I
Proposed Lots of Shortening

Lot	Stability A.O.M.	Iodine Value
	<i>hrs.</i>	
1C.....	20	90-95
2C.....	40	75
3C.....	60	67.5
4C.....	90	62.5
5C.....	150	60.0
6C.....	250	57.5
1S.....	50	78
2S.....	150	70
3S.....	250	65

Preparation of Shortenings and Biscuits

Eleven lots of shortening of varying stability values were prepared from a common lot of three basic shortening materials: i.e., lard, cottonseed oil, and extracted soybean oil. Manufacturing procedures were controlled, as far as possible, to minimize variables. The lard was prepared by dividing a batch of refined steam lard, one half of which was stabilized with 0.01% N.D.G.A. (nordihydroguaiaretic acid); the other half was utilized as a control. The crude oils were alkali refined (batch process) and bleached with Super Filtrol. The cottonseed and soybean oil shortenings were made from homogeneous batches of the respective liquid oils drawn from a hydrogenator at successive stages in the hydrogenation operation. Sufficient hydrogenated cottonseed oil flakes were added to the softest lots before plasticizing to provide practical workability during the mixing of the ingredients prior to baking. Each lot of hydrogenated oil was refined, deodorized, and plasticized under identical conditions, utilizing pilot plant equipment. Table I indicates the proposed spread in accelerated stability values of the cottonseed oil (C) and soybean oil (S) shortenings. For the purpose of this experiment, approximate iodine values, as indicated, were predetermined as a means of controlling the hydrogenation.

The vegetable oil shortening and lard were packaged in 37-pound hermetically sealed containers for the bakery tests and held under refrigeration prior to use. A detailed chemical examination was completed on each lot of shortening as well as on the refined, bleached vegetable oils prior to hydrogenation. The Type IV biscuits, except those in which hydrogenated soybean oil was incorporated, contained 2% of commercial soy lecithin. The Type IV series also contained a higher fat content as well as other ingredient variables. Biscuits were prepared in accordance with Army specification for ration biscuits (6). Each of the 11 variable shortenings was incorporated in Type I and Type IV ration biscuit formu-

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³ Since the evaluation of the degree and seriousness of "reverted" flavors from soybean oil shortening was a problem of consumer acceptance, the third objective will not be treated in these papers. It will, however, be reported separately by investigators of the Food Acceptance Branch of the QM Food and Container Institute for the Armed Forces who were assigned to this aspect of the program.

las. Table II shows the basic ingredients⁴ used and the actual batch size. The mixing of the ingredients, baking, and packaging were carried out in a modern commercial bakery according to good commercial practice.

TABLE II
Biscuit Formulas (Batch Size)

Material	Type I	Type IV
	lbs.	lbs.
Flour, Soft Strength.....	340	400
Flour, Graham.....	60	None
Sodium Bicarbonate.....	2 1/4	1 7/16
Ammonium Bicarbonate.....	2 1/2	None
Vicream (mono-calcium phosphate).....	2 3/8	1 7/8
Sugar.....	20	18
Invert Sugar Syrup.....	18	6
Shortening.....	50	76
Non-fat Dry Milk Solids.....	None	16
Dried Yeast.....	4	6
Salt.....	5	5
Lecithin.....	None	1 9/16*
Topping Salt (Approximate).....	None	1†
Water.....	As required	As required

* Not included in biscuits containing hydrogenated soybean oil shortening.

† Per 100 lb. finished biscuits.

Three types of packaging conditions (cartons, sealed cans, and punched cans) were utilized for each of the 22 lots of ration biscuits. The punched cans (sealed cans with a 2-mm. hole in the top) were included in this experiment in order to compare conditions of storage involving a limited and a free supply of atmospheric oxygen. The cartons and cans were of the type specified for Army ration biscuits (6).

The biscuits were stored at 70° and 100°F. The humidity of the constant temperature room was not controlled. Examinations of biscuits were scheduled for 1-, 2-, 4-, 6-, 9-, 12-, 15-, 18-, 21-, and 24-month intervals.

Initial Analyses of Shortening

Representative samples of the 11 shortenings were shipped via refrigerated express to the Eastern Regional Research Laboratory which conducted all of the physical and chemical analyses except the trace metal assays. The results of the initial shortening examinations are tabulated in Tables III and IV; calculated data related to the glyceride composition are presented in Table V.

Methods of analysis for the shortening were as follows:

Active Oxygen Method (4). Swift Stability apparatus, using a temperature of 98.5°C. Endpoint of induction period of the vegetable shortenings was recorded as the time required to produce a peroxide value of 100 milliequivalents per kilogram of sample; the endpoint of the lard samples was the time required to produce a peroxide value of 20 milliequivalents per kilogram of sample.

Oxygen Absorption Method (7). Barcroft-Warburg apparatus, using static method at 100°C. Endpoint of the induction period of vegetable shortenings was recorded as the time required for the absorption of oxygen equivalent to 3 grams per kilogram of sample, and for lard the time required for the absorption of 1 gram of oxygen per kilogram of sample.

Iodine Value. Standard Wijs Method (5).

Thiocyanogen Value. Method of Riemenschneider, Swift, and Sando (8), using 0.1 N solution, 24-hour absorption period but increasing carbon tetrachloride to 27.4%.

Melting Point. Official A.O.A.C. Wiley Method (5).

Copper. According to the method of Bendix and Grabenstetter (1).

Iron. According to the method of Thompson (10).

Nickel. According to a modified dimethylglyoxime procedure (9) employing acid extraction of the sample (2).

The copper, iron, and nickel contents of the shortening (Table III) are quite low and fairly uniform.

Organoleptic Observations. Odor and flavor observations were made at room temperature on the freshly opened samples. Also, odor scoring was carried out on a) a melted and thoroughly mixed 20-gram sample in a covered 40 x 50-mm. weighing bottle maintained at 55°C. during the test by means of thermally controlled hotplate, and b) a sample, as in a), heated in an oven to 400°F., removed and allowed to cool to room temperature. Sample held in dark for 24 hours and then heated and held at 55°C. during the organoleptic testing.

TABLE III
Initial Shortening Analyses (General)

	Stability		Wiley	Metals			Organoleptic Observations*	
	A.O.M. O ₂ -Abs.		M. P.	Cu	Fe	Ni	Odor Flavor	
	hrs.	hrs.	°C.	p.p.m.	p.p.m.	p.p.m.		
Lard								
1L.....	4.0	1.5	37.7	0.15	0.90	0.10	3	3
2L.....	18.0	9.6	37.6	0.05	1.90	0.05	3	3
Cottonseed								
Oil.....	8.5	7.0
1C.....	20.0	13.7	45.6	0.25	0.30	0.65	1	1
2C.....	38.5	23.0	43.5	0.10	0.60	0.85	1	1
3C.....	78.0	42.2	37.4	0.15	0.10	0.80	1	1
4C.....	132.0	81.0	39.5	0.05	0.50	0.85	1	1
5C.....	173.0	105.3	40.9	0.15	0.60	0.85	1	1
6C.....	272.0	150.0	42.7	0.05	0.15	0.80	1	1
Soybean								
Oil.....	7.0	6.7
1S.....	48.0	29.8	52.6	0.00	0.10	0.05	1	1
2S.....	170.0	91.5	42.1	0.15	0.55	0.05	2	2
3S.....	291.0	138.0	45.6	0.05	0.30	0.05	2	2

* Code: 1—Good, bland. 2—Satisfactory, trace of reversion; 3—Satisfactory, stale, meaty.

Chemical analyses made on the 22 lots of biscuits consisted of the following:

Moisture. Official Vacuum Oven Method of the A.O.A.C. (5).

pH. Slightly modified Electrometric Method of the A.O.A.C. (5), using a buffer solution of pH 7.0 for standardization.

Iron Content. Obtained according to the method of Thompson (10).

Copper Content. Determined by the method of Bendix and Grabenstetter (1).

Peroxide Value. Transfer approximately 10 grams of ground sample into a 250-ml. Erlenmeyer flask and add 50 ml. of chloroform. Extract the fat at room temperature for one-half hour, occasionally shaking. Filter through a Whatman No. 43 filter paper, previously washed with chloroform, and collect the filtrate in a small flask.

Pipette a 20-ml. aliquot of the filtrate into a 250-ml. Erlenmeyer flask, add 30 ml. of glacial acetic acid and 0.5 ml. of a saturated solution of potassium iodide. Mix by swirling and allow to stand for exactly two minutes. Add 30 ml. of distilled water and immediately titrate the liberated iodine with 0.01 or 0.1 N sodium thiosulfate, using starch as an indicator. Determine the weight of fat in the aliquot by pipetting a 5-ml. portion of the filtrate into a dried, tared beaker, evaporating off the solvent on a steam bath

⁴ All ingredients were drawn from common lots.

TABLE IV
Initial Shortening Analyses (Spectrophotometric)

	Iodine No.	S.C.N. Value	Spectrophotometric Data					
			Lino-leic	Lino-lenic	Arachi-donic	Conj. Diene	Conj. Triene	Conj. Tetr.
Lard								
1L.....	68.8	57.5	12.1	0.90	0.40	0.22	0.007	.000
2L.....	68.8	57.5	12.0	0.92	0.40	0.20	0.001	.000
Cottonseed Oil.....		64.3	50.5	0.79	0.00	0.32	0.12	0.004
1C.....	106.3	57.8	35.1	0.28	0.00	0.32	0.03	0.000
2C.....	88.7	58.8	16.8	0.07	0.00	0.36	0.00	
3C.....	74.9	59.4	6.9	0.00	0.00	0.19	0.00	
4C.....	67.4	58.0	3.3	0.00	0.00	0.13	0.00	
5C.....	62.5	56.8	2.2	0.00	0.00	0.11	0.00	
6C.....	60.4	54.5	1.0	0.00	0.00	0.09	0.00	
Soybean Oil.....		82.4	53.3	7.6	0.00	0.20	0.03	0.004
1S.....	134.8	68.5	7.5	0.43	0.00	0.16	0.00	0.000
2S.....	80.2	64.6	2.1	0.00	0.00	0.12	0.00	
3S.....	68.6	60.4	0.91	0.00	0.00	0.08	0.00	
	63.0							

and drying the beaker and contents in an air oven at 100°C. to constant weight.

$$\text{Peroxide value} = \frac{\text{ml. of Na}_2\text{S}_2\text{O}_3 \times N \times 1000}{\text{wt. of fat in 20-ml. aliquot}}$$

Rancimeter Accelerated Stability Test. The method of James (3) for cereal products was modified to adapt it to Army ration biscuits. It was necessary to increase the aeration temperature. A temperature of $216 \pm 0.5^\circ\text{F}$. was selected, necessitating the use of a light mineral oil bath, more adequate means of circulation, and more sensitive temperature control than originally employed. Inasmuch as these modifications have necessitated considerable alteration of the original method, a general description of technique, with special attention to modifications, is presented.

Sample Preparation. Grind the biscuits in a Waring Blendor. Take that portion which passes a No. 12 sieve (Tyler-Equivalent) but does not pass a No. 20 sieve. The sieves should be made of stainless steel. Place 30 grams of the prepared sample in the aeration tube. Place the surgical cotton in each end to hold the sample firmly in place. Prepare three aeration tubes for each sample on test and a blank aeration tube which contains cotton only.

Determination of Rancimeter Values. Place the prepared aeration tubes in instrument and connect in the usual manner. Arrange the outlet of each discharge tube so that the effluent air will pass into a 25 x 150-mm. test tube containing 10 ml. of 0.005 N KMnO_4 and 1 ml. H_2SO_4 (1-4).

Adjust the air flow through each tube so that between 180 and 200 bubbles per minute flow through the reaction tubes. Maintain this rate throughout the

test. Correct for slight variation by means of the screw clamps located on the air supply tubes.

Remove the reaction tubes containing the permanganate at exactly one-hour intervals and replace with tubes containing fresh reagents. Transfer quantitatively the contents of the tubes, which have been removed from the instrument, into 125-ml. Erlenmeyer flasks. Add 5 ml. of 5% potassium iodide (prepared daily) to each flask and titrate with 0.005 N sodium thiosulfate, using starch indicator. The difference between the blank titration and the sample titration equals the ml. of 0.005 N KMnO_4 reduced.

Plot the ml. of reduced permanganate against the aeration time in hours. The stability or induction time is defined as the number of hours required to reduce 3 ml. of 0.005 N KMnO_4 . This value, although beyond the actual end of the induction period, has been found to be more reproducible since biscuits of long stability tend to exhibit only a gradual but somewhat erratic increase in the amount of permanganate reduced per hour at the end of the induction period. At a value of 3 ml. of reduced permanganate the curve exhibits a definite slope and plotted values are more readily interpolated.

As it requires approximately 2 hours for the samples to come to temperature when placed in the instrument, overnight removal will result in high values. Continuous aeration can be obtained by starting one tube (pilot) 24 hours in advance of 2 replicate tubes. The latter need not be titrated until the pilot tube has reached or passed the end of the induction period. Overnight operation may be accomplished by aeration into a reaction tube filled to the mark with distilled water. Frequently, only values beyond the defined end of the induction period will be obtained for the

TABLE V
Calculated Glyceride Composition

	Comp. by I.V.-T.V. Calculations			Comp. by Iodine-Spectrophotometric Calculations			
	% Oleic	% Linoleic	% Sat.	% Oleic	% Linoleic	% Linolenic	% Sat.
Lard							
1L.....	53.2	13.2	33.5	51.1	12.9	0.94	35.1
2L.....	53.2	13.3	33.5	51.2	12.8	0.96	35.0
Cottonseed Oil.....	22.5	50.2	27.3	14.7	53.1	1.0	32.2
1C.....	28.9	36.9	34.3	27.5	37.1	0.3	35.1
2C.....	48.7	19.1	32.2	50.4	18.0	0.1	31.5
3C.....	59.6	9.3	31.1	63.5	7.4	0.0	29.1
4C.....	62.5	5.0	32.6	65.3	3.6	0.0	31.1
5C.....	62.2	4.0	33.8	65.3	2.4	0.0	32.3
6C.....	61.0	2.5	36.5	63.8	1.1	0.0	35.1
Soybean Oil.....	(30.5)	(62.6)	(6.9)	31.2	56.0	8.0	4.8
1S.....	65.7	13.7	20.6	75.4	8.1	0.45	16.0
2S.....	70.9	4.4	24.7	75.0	2.3	0.0	22.7
3S.....	67.7	2.8	29.6	72.3	1.0	0.0	26.7

TABLE VI
Copper and Iron Content of Biscuit Ingredients

Formula Ingredient	Cu. p. p. m.	Fe. p. p. m.
Flour.....	1.6	6.0
Flour, Whole Wheat.....	4.4	35.3
Salt.....	0.14	7.4
Soda.....	0.14	3.5
Sugar.....	0.7	1.0
Skim Milk.....	3.4	4.4
Syrup Invertase.....	0.9	9.7
Vicream (Mono-calcium phosphate).....	3.4	2.2
Yeast.....	148.6	94.2

pilot tube; however, interpolation of the curve is possible after the slope of the curve is established with the follow-up tubes. Results are reported as the average of triplicate determinations. Fig. 1 shows typical plots of Rancimeter data.

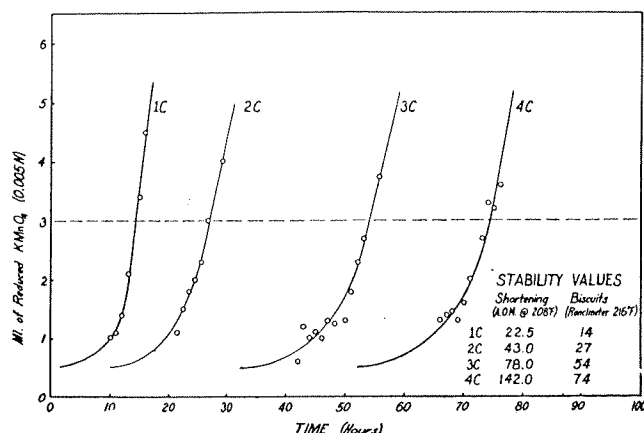


FIG. 1. Typical Rancimeter curves. Type IV biscuits containing hydrogenated cottonseed oil of varying A.O.M. values.

Copper and iron determinations were made on the individual biscuit ingredients (Table VI) and on each lot of the finished biscuits (Table VII). The copper and iron contents of the biscuits are quite uniform and represent an average increase, due to water added and to processing contamination, of only 1.0 and 3.3 p.p.m., respectively, for Type I biscuits and 0.6 and 2.5 p.p.m., respectively, for Type IV biscuits.

A few of the lots of shortenings initially demonstrated the presence of some peroxides, but these were apparently destroyed as a result of thermal decomposition during the baking process. The variation observed in the moisture contents of the various lots of biscuits are perhaps indicative of commercial manufacturing procedure. It does present a variable which was not controlled.

Discussion of Accelerated Stability Tests

The results of the accelerated stability tests on the shortenings and biscuits are presented in Table VIII. The Swift Stability values (A.O.M.) are the results obtained by three collaborating laboratories. All subsequent reference to these data will refer to the mean values only.

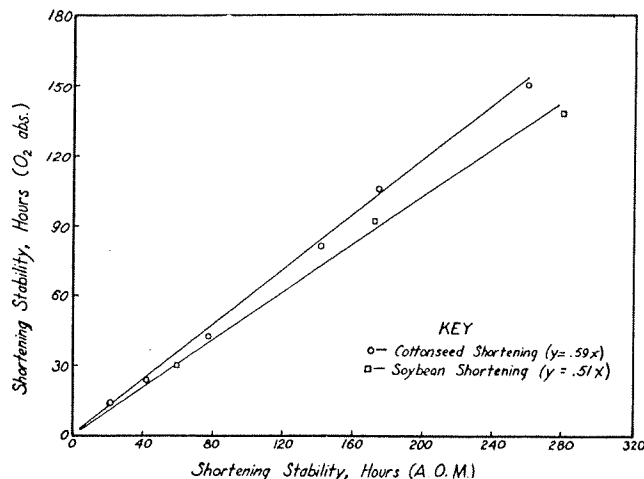


FIG. 2. Relationship of shortening accelerated stability values as determined by active oxygen and oxygen absorption methods.

The correlation of the A.O.M. with Oxygen Absorption data obtained on the vegetable oil shortenings is best shown graphically (Fig. 2). It can be observed that a linear relationship exists. The Oxygen Absorption Method tends to give slightly lower values for the soybean shortenings than for the cottonseed shortenings when based upon identical A.O.M. values.

The relationship of the accelerated stability values of the shortening (A.O.M.) and the biscuits (Rancimeter) is shown in Fig. 3. These curves show the effect of shortening stability on biscuit stability as well as variations of ingredients of two different biscuit formulas. The differences noted between Type I and Type IV biscuits containing cottonseed shortening probably can be attributed to the addition of 2% (shortening basis) commercial soybean lecithin to the Type IV biscuit. No significant difference was observed when lecithin was omitted from the biscuit formulas containing hydrogenated soybean oil shortenings.

The cottonseed shortening curves would tend to substantiate Q.M.C. specification for 100-hour biscuit shortening since this value is in the region beyond

TABLE VII
Initial Analyses of Biscuits

Lot	TYPE I					TYPE IV				
	Moisture	pH	Cu.	Fe.	P. V.	Moisture	pH	Cu.	Fe.	P. V.
	%		p. p. m.	p. p. m.	me/ 1000 g.	%		p. p. m.	p. p. m.	me/ 1000 g.
1L.....	5.4	6.6	4.7	15.4	0.0	4.3	6.5	5.3	12.7	0.0
2L.....	4.9	6.6	3.4	15.3	0.0	4.6	6.6	3.2	11.2	0.0
1C.....	4.7	6.7	3.7	13.1	0.0	4.2	6.6	3.4	12.0	0.0
2C.....	5.1	6.7	3.5	12.5	0.0	4.4	6.6	3.6	10.6	0.0
3C.....	5.8	6.8	4.0	12.8	0.0	5.7	6.6	3.1	10.0	0.0
4C.....	6.8	6.8	3.7	15.1	0.0	6.1	6.6	3.0	10.1	0.0
5C.....	5.8	6.8	3.9	13.8	0.0	5.7	6.6	3.8	9.9	0.0
6C.....	5.3	6.7	3.8	14.7	0.0	3.7	6.5	3.9	11.0	0.0
1S.....	3.9	6.6	4.0	12.0	0.0	5.4	6.7	4.2	12.5	0.0
2S.....	5.5	6.7	3.9	14.3	0.0	3.6	6.5	3.9	10.5	0.0
3S.....	5.7	6.8	4.4	14.0	0.0	5.9	6.6	4.5	10.5	0.0

TABLE VIII
 Accelerated Stability Values of Shortenings and Biscuits

Lot	Shortenings					O ₂ Abs. 100°C.	Biscuits	
	A.O.M. (98.5°C.)						Rancimeter (216°F.)	
	A	B	C	Mean	Type I		Type IV	
	hrs.	hrs.	hrs.	hrs.	hrs.	hrs.	hrs.	
1L.....	4	5	5	1.5	16.5	15.5	
2L.....	18	24	21	9.6	22.0	19.0	
1C.....	20	25	22	22	13.7	14.0	14.0	
2C.....	39	45	37	40	23.0	25.5	27.5	
3C.....	78	80	77	78	42.2	29.5	55.0	
4C.....	132	142	155	143	81.0	51.0	72.0	
5C.....	173	175	179	176	105.3	53.0	78.5	
6C.....	272	244	275	264	150.0	62.0	82.5	
1S.....	48	70	60	59	29.8	35.5	35.5	
2S.....	170	180	226 ¹	175	91.5	81.0	82.5	
3S.....	291	265	294	283	138.0	106.0	106.0	

¹ Not averaged.

which greater shortening stability results in only a slight increase in biscuit stability.

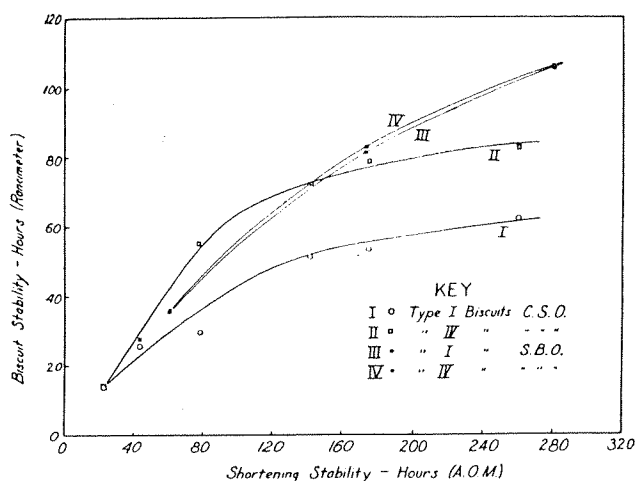


Fig. 3. Relationship of biscuit and shortening accelerated stability values.

Thus there would appear to be little gained by using cottonseed shortening above a 100-hour accelerated stability value, particularly since incorporating shortening in the biscuit dough becomes more difficult with additional hydrogenation. In every instance an increase in Rancimeter values follows an increase in shortening stability. A more direct relation, however, is observable in biscuits containing soybean oil shortening.

The Rancimeter values for the Type IV biscuits containing lard (1L) and lard plus N.D.G.A. (2L) (Table VIII) are somewhat lower than those obtained for the corresponding Type I biscuits, indicating that lecithin incorporated in the biscuits decreased their stability when lard was the shortening. Evaluation of accelerated stability tests by means of actual storage studies will be discussed in a second paper in which it will be shown, in general, that the storage studies validate the results obtained by the accelerated tests. It is not to be construed, however, that a given number of hours by either test will accurately predict the shelf life of that product. Variable conditions of packaging, relative humidity, temperature, etc., make this impossible. Storage tests on the products under controlled conditions of packaging, humidity, and storage temperature are required for interpreting accelerated results. With such information at hand accelerated methods become useful in maintaining quality control.

Summary

An experiment involving the commercial production and packaging of two types of Army ration biscuits prepared from common ingredients with nine lots of vegetable oil shortening of increasing stability values and two lots of lard as the only ingredient variables has been described, and the initial analytical data presented.

Evaluation of accelerated stability tests on both shortenings and biscuit shows that:

1. A linear relationship exists between Swift Stability (A.O.M.) and oxygen absorption (Warburg) values obtained on cottonseed oil and soybean oil shortening.

2. Increasing the accelerated stability values of the shortening by additional hydrogenation of the vegetable oils resulted in greater accelerated stability values for the biscuits containing the corresponding shortenings. A nearly direct relationship was found between the stability of soybean oil shortenings (Swift Stability Values) with the stability of the corresponding biscuits (Rancimeter). In the case of the cottonseed oil shortenings the increase in biscuit stability was marked to about a 100-hour accelerated stability value, but was much less pronounced above 100 hours.

3. Addition of commercial lecithin increased the stability values of the biscuits containing cottonseed oil shortenings, but had an adverse effect on the stability of biscuits containing lard and lard plus N.D.G.A.

Results obtained from examinations of biscuits packaged in fiberboard cartons, punched cans, and sealed cans and stored for two years at 70°F. and 100°F. are contained in the second paper of this series.

Acknowledgment

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